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Attorney's Docket No.:

774-010234-US(PAR)

#### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: OOI et al.

Group No.:

Serial No.: 09/802,084

Filed: 3/08/01

For: QUANTUM WELL INTERMIXING

Examiner:

**Commissioner of Patents and Trademarks** 

Washington, D.C. 20231

#### TRANSMITTAL OF CERTIFIED COPY

Attached please find the certified copy of the foreign application from which priority is claimed for this case:

Country

: Singapore

Application Number

: 200004787-8

Filing Date

: 11 September 2000

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Date of Filing

11 SEPTEMBER 2000

Application Number

: 20004787-8

Applicant(s)

: NANYANG TEHNOLOGICAL UNIVERSITY

Title of Invention

MULTIPLE WAVELENGTH LASERS

CHIG KAM TACK
Assistant Registrar
for REGISTRAR OF PATENTS
SINGAPORE

#### SINGAPORE THE PATENTS ACT (CHAPTER 221) THE PATENTS RULES

200004787-8

The Registrar of Patents, Registry of Patents.

1 1 SEP 2000

#### REQUEST FOR THE GRANT OF A PATENT

THE GRANT OF A PATENT IS REQUESTED BY THE UNDERSIGNED ON THE BASIS OF THE PRESENT APPLICATION.

I.	Title of Invention		MULTIPLE WAVELENGTH LASERS
II.	Applicant(s) (see note 2)	(a) Name	NANYANG TECHNOLOGICAL UNIVERSITY
	(000 11010 2)	Body Description/ Residency	
		Street Name & Number	SCHOOL OF ELECTRICAL AND ELECTRONIC ENGINEERING
1		City	NANYANG AVENUE
'		State	
1		Country	SINGAPORE 639798
		(b) Name	-
		Body Description/	•
		Residency	
		Street Name & Number	
		City	
	£ 42	State	
		Country	
,		(c) Name	-
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III. Declaration of	Country/		File no.					
Priority	Country	•						
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IV. Inventors								
(see note 4)								
*								
(a) The applicant(s) is/are the	Yes	X	No					
sole/joint inventor(s)								
	X Yes		No	•				
(b) A statement on Patents								
Form 8 is/will be furnished.								
V. Name of Agent (if any)	HAQ & NAMAZIE P	ARTNERSHIP						
(see note 5)	-							
VI. Address for Service	Block/Hse No.		Level No.	1				
(see note 6)	BIOCKITSE NO.	1	Level IVO.					
	Unit Ne/PO Box	765	Postal	901515				
			Code					
	Street Name	Robinson Road Post Office						
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\	Building Name			· ·				
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VII. Claiming an earlier filing date	Application No.	T'						
under section 20(3),26(6) or	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,							
47(4).		<del></del>	1					
(see note 7)	Filing Date							
	[Please tick in the relevant space provided]:							
	( ) Proceeding under rule 27(1)(a).							
	Date on which the earlier application was amended =							
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VIII. Invention has been displayed at an International Exhibition (see note 8)		Yes	X No				
IX. Section 114 requirement (see note 9)	The invention relates to and/or used a micro-organism deposited for the purposes of disclosure in accordance with section 114 with a depositary authority under the Budapest Treaty.  Yes  X						
X. Check List	A. The application cor			per of			
(To be filled in by applicant or agent)	sheet(s):-  1. Request 2. Description 3. Claim(s). 4. Drawing(s). 5. Abstract. B. The application as 1. Priority document 2. Translation of prio 3. Statement of Invent 4. International Exhibit	ority docume ntorship & ri	ent ight to grant		s s		
XI. Signature(s) (see note 10)	Applicant (a)	hy	2				
*	Date Applicant (b)	11 SEPT.	2000				
-	Date Applicant (c)						
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- 6. An address for service in Singapore to which all documents may be sent must be stated at paragraph VI. It is recommended that a telephone number be provided if an agent is not appointed.
- 7. When an application is made be virtue of section 20(3), 26(6) or 47(4), the appropriate section should be identified at paragraph VII and the number of the earlier application or any patent granted thereon identified.
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MH/JN/jp/5000811SGF

#### MULTIPLE WAVELENGTH LASERS

#### Field of the Invention

The present invention relates to laser structures for providing plural different radiation wavelengths and to a method of manufacturing such lasers.

#### Background of the Invention

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The growth of Internet traffic, multimedia services and high-speed data services has exerted pressure on telecommunication carriers to expand the capacity of their networks quickly and cost effectively. The three options normally available are:

- 1) Install new fibers,
- 15 This has problems due to costs and difficulties of rights of way.
  - 2) Increase the bit rate of the transmission system.

This option has inherently limited growth potential.

3) Employ wavelength division multiplexing.

This third option allows manifold increase of the network capacity at a modest cost.

Wavelength division multiplexing, also known as frequency division multiplexing, allows better use of the available bandwidth by carrying plural optical transmission channels over a single fiber. Each channel operates at a different frequency/wavelength. Alternatively data may be sent in packets each at different frequencies or wavelengths.

Wavelength division multiplex transmission systems may also enable longer span distance between repeaters, and more spans before regeneration is needed. This makes such systems attractive for long haul transmission.

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The heart of the wavelength division multiplexing systems multiple-wavelength laser device. In known commercial devices using distributed feedback lasers, the laser wavelength may be tuned by defining gratings with a submicron period to perform frequency chirp. The device grating is conventionally formed by electron lithography, as holography has not been found suitable for creation of complex multi-pitch grating structures. Electron beam writers are however costly and of low electron beam lithography throughput. Thus device production. favourable for large-scale addition, regrowth of the sample is necessary, and this entails additional cost.

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Alternatively selective area epitaxial growth has been 20 lasers. fabricate multiple wavelength This utilizes differences in epitaxial technique layer composition and thickness produced by growth through a mask to achieve spatially selective bandgap variation. This process works well under a precisely controlled set 25 of parameters but is difficult to manipulate in a generic fashion.

Each of the methods of the prior art has disadvantages.

For example, using etching and regrowth methods results in poor optical confinement to the wave-guide devices as a result of sidewall inhomogeneity. As it involves

multiple processing steps there is often a low yield and throughput may be low.

Selective area epitaxy is also a complicated technique and requires complex steps of sample preparation. Also, the technique may give non-uniform growth rate across the strip that prevents subsequent planar processing. For the same reason, passive wave-guide sections may be relatively lossy.

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Quantum well intermixing can be used selectively to modify the bandgap energy of a quantum well sample. However, spatial control of the bandgap using the prior art quantum well intermixing techniques is complicated. For example using varying thickness of silicon dioxide layers as intermixing sources of impurity-free vacancy disordering, orimplant masks for impurity induced disordering (IID) increases the number of processing steps of lithography and deposition of dielectric caps.

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To overcome this complexity the prior art includes a one-step spatially controlled quantum well intermixing technique based upon impurity-free vacancy disordering. In this process, the semiconductor is patterned with very small areas of  $SrF_2$  followed by coating the sample with  $SiO_2$ . The degree of intermixing then depends on the area of semiconductor substrate in direct contact with the  $SiO_2$  layer. This technique, although being one-step, requires electron beam lithography, which decreases the process throughput and increases design complexity.

It is accordingly an object of the present invention to at least partially mitigate the difficulties of the prior art.

#### 5 Summary of the Invention

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According to one aspect of the invention there is provided a laser structure, said structure comprising a plurality of quantum well regions on a semiconductor substrate, each said region containing a respective different concentration of an impurity, to thereby provide a corresponding plurality of different radiation wavelengths in use.

Embodiments of the invention include monolithically integrated multiple-wavelength laser structures in 1.55  $\mu \rm m$  GaInAs/GaInAsP materials. Typical embodiments have more than 5 channels and are produced using a low energy IID process.

- According to a further aspect of the present invention 20 there is provided a method of manufacturing a photonic integrated circuit comprising providing a having a quantum well region, and performing quantum well intermixing, characterized in that said step. of well intermixing comprises 25 performing quantum differentially masking portions οf said implanting impurities into said differentially masked portions and annealing said structure.
- 30 Preferably said step of differentially masking portions of said region comprises forming a masking layer on said

portion of said region and differentially etching said masking layer.

Advantageously before said etching step the method comprises forming a photoresist on said masking layer, applying masks having different optical densities to said photoresist and developing said photoresist.

Conveniently said step of forming a photoresist may comprise spin-coating said photoresist on said masking layer.

Conveniently said masking layer comprises a dielectric such as silicon dioxide. Alternatively said masking layer may comprise a polymer or a metal.

Preferably said etching step comprises dry etching.

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Advantageously said dry etching has substantially a oneto-one selectivity between photoresist and said masking layer.

25 Conveniently process parameters of the said dry etching system are selected to provide said selectivity.

Conveniently said annealing step is performed in a rapid thermal processor, alternatively a rapid thermal annealer or furnace may be used. Preferably said impurities comprise phosphorus and arsenic.

#### 5 Brief Description of the Drawings

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An exemplary embodiment of the invention will now be described with reference to the accompanying drawings in which: -

10 Figure 1 shows in diagrammatic form a typical quantum well structure used in the present invention;

Figure 2 shows an exemplary process flow for fabrication of multiple wavelength lasers;

Figure 3(a) and (b) show the thickness of resist and oxide before and after reactive ion etching;

Figure 4(a) to (c) show the results of measurements on the first embodiment of the invention and;

Figure 5(a) to (c) shows the results of measurements on the second embodiment.

#### 25 <u>Description of the Preferred Embodiments</u>

In the various figures, like reference numerals refer to like parts.

30 Referring to Figure 1, an exemplary semiconductor structure suitable as the basis for manufacturing multiple wavelength lasers was formed. An  $InP/Ga_{1-x}$ 

In,As/Ga, In,As,P, structure (10) was grown using metal organic chemical vapour deposition (MOCVD) on an InP substrate. An active region was formed, comprising a single undoped quantum well region consisting of a 5.5 nm wide GaInAs quantum well (14), and 12 nm GaInAsP  $(λ_c=1.26μm)$  barriers (22,24). The active region was bounded by step or graded index (GRIN) GaInAsP confining layers (20-21, 25-26). The confining layers comprise 50nm of material having  $\lambda_{\alpha} = 1.18~\mu m$  and 80nm of material having  $\lambda_{\alpha}=1.05$  µm, respectively. The structure was lattice matched to InP throughout, and was completed with a 1.4 µm InP upper cladding layer (28) and a layer (30) of 0.65 µm GaInAsP followed by a 0.1µm GaInAs layer (32) forming a contact layer. The lower cladding layer (12) was Sulphur-doped to a concentration of 2.5x10<sup>18</sup> cm<sup>-3</sup>. upper cladding layer (28) was doped with Zn to concentration of 7.4x1017 cm-3 and the subsequent layers (30,32) were doped with  $2x10^{18}$  cm<sup>-3</sup> and  $1.3x10^{19}$  cm<sup>-3</sup> concentration of Zn respectively. The core of the structure, forming a wave-guide, was undoped, forming a P-I-N structure with its intrinsic region restricted to the quantum well and GRIN layers.

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Referring now to Figure 2, an outline of the process of the invention will now be given: -

Two embodiments of the invention are provided. In the first, a quantum well structure (such as that described with respect to Figure 1) was implanted with As impurities that varied in amount spatially by virtue of implantation through a varying thickness gray mask. In

the second, a similar type of mask was used to implant P ions in a similar way.

Simulations of the impurity implantation ranges and vacancy distribution were first carried out to determine the thickness of SiO<sub>2</sub> required for the bandgap shift that was sought. It would alternatively be possible to use other theoretical calculations or empirical approaches.

10 The thickness of SiO<sub>2</sub> mask required to totally block As ions from reaching the semiconductor during implantation was found to be around 400 nm. The thickness of SiO<sub>2</sub> mask required to totally block P ions from reaching the semiconductor during implantation was found to be around 900 nm.

In step (i) laser isolation and alignment mark etching is performed, for example using a first mask having a  $20\mu m$  stripe pattern. An exemplary etching process is a wetetching process using sulphuric acid, hydrogen peroxide and water in 1:8:40 ratio whereby  $0.15\mu m$  of the GaInAs and GaInAsP contact layer is removed.

After the laser isolation and alignment mark etching step the structure is coated with oxide to the above respective thickness according to the impurity to be used, to act as a mask.

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It will be understood that masks other than oxide could be used, for example, polymer, metal or photoresist mask materials, or dielectrics other than oxide.

Next, a positive photoresist is spin-coated onto the structure.

In step (ii), photolithography is carried out to transfer the gray patterns onto the structure: -

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The gray mask technique of the invention makes use of different transparencies of masks to control the degree of the exposure of photoresist at selected regions and thickness photoresist of different development. The degree of the development of photoresist after UV exposure has a linear relationship with the optical density. In the current embodiments, gray mask was selected having 10 levels, from 0.15 to 1.05 with a step of 0.1, of optical density, see Figure 4. By this means, it was intended to provide 10 different bandgaps after quantum well intermixing. It will of course be clear to those skilled in the art that fewer or greater numbers of bandgaps could be provided, according to the requirements of the application.

The relationship between optical density of mask and the UV light transmissivity level during the lithography process can be expressed using the following equation.

OD = -log(T)

where OD is optical density and T is transmissivity.

In step (iii), a dry etching process is performed so as to etch correspondingly into the oxide mask, so that different thicknesses of oxide are produced in correspondence with the gray masking.

For the embodiments, a reactive ion etching process with 1:1 selectivity between photoresist and SiO<sub>2</sub> was chosen as the dry etching process. This process was performed in a conventional parallel plate radio-frequency reactive ion etching system using CF<sub>4</sub> and O<sub>2</sub> as process gases. Statistical methods were then used to optimise the parameters of this novel non-selective reactive ion etching process. To achieve 1:1 non-selective etching, the following settings were found suitable for the particular materials in question: pressure of 95~120 mTorr, RF power 40~100 W, CF<sub>4</sub> flow rate 20~80 standard cubic centimetre per minute (sccm) and O<sub>2</sub> flow rate 1~10 sccm.

The thickness of the resist and SiO2, as measured from a 15 surface profiler, both before and after reactive-ion etching for the two embodiments are given in Figure 3a and 3b. The first embodiment (As\* implanted -Fig. 3a) shows a slightly inferior quality of gray level transition; also saturation of pattern thickness at both 20 the upper and the lower density regions are detected. This effect is mainly due to the thin resist required for imperfect the As\*\* implanted sample, which causes imperfect after development. With the gradation gradation, the SiO<sub>2</sub> pattern profile is directly affected. 25

However, for the second embodiment (P\*\* implanted-Fig. 3b) smooth and obvious transitions at each density levels are obtained.

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In step (iv), implantation is carried out with impurity ions through the mask. In the embodiments, the samples

were implanted with  $1 \times 10^{14}$  cm<sup>-2</sup> at 350~500 keV for both As and P impurities, at  $150\sim230$  °C. The result of the implantation through the masks is that the degree of implantation is inversely proportional to the thickness of the graded masks.

In step (v), quantum well intermixing step was carried out by annealing using a rapid thermal processor, at 500~800 °C for about 80~120 s along with the SiO<sub>2</sub> implant mask intact. The SiO<sub>2</sub> implant mask was removed after quantum well intermixing.

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After this, laser fabrication was completed as follows: -

- a) front contacts (p-type: Ti/Au, 50 nm/200 nm) were metallised using an electron beam evaporator.
- b) the samples were then thinned to a thickness of around 180 $\mu m$ .
- c) a metallisation for the back contact (n-type: Au/Ge/Au/Ni/Au, 14nm /14nm /14nm /11nm /200nm) was performed by evaporation;
- d) a metal lift-off step for the laser isolation trench was performed. The heavily doped GaInAs and GaInAsP contact layers were removed at the trenches to achieve good electrical isolation between lasers.
- 25 e) the whole fabrication was completed by annealing the samples using rapid thermal processing at 300~450 °C for 45~100 s.

The lasers were then cleaved for testing. Each individual laser has a dimension of 400 x  $500\mu\text{m}^2$  and  $50\mu\text{m}$  width of active window,  $500\mu\text{m}$  cavity length and  $20\mu\text{m}$  width of isolation trench.

In step (vi), the testing step, intensity vs. current and spectrum measurements were carried out. Each laser was pumped individually during the characterisation and measurements.

A linear correlation between thickness of implant mask and the wavelength emission was found in Figures 4a and 5a. This further verifies the linear relationship between the degree of point defects generated with different thickness of implant mask and the degree of intermixing or bandgap-tuning.

As shown in Fig 4b, in the first embodiment, using As impurities, a 6-channel monolithic multiple wavelength laser structure was achieved. The wavelengths were 1.555  $\mu$ m, 1.544  $\mu$ m, 1.535  $\mu$ m, 1.518  $\mu$ m, 1.503  $\mu$ m, and 1.484  $\mu$ m respectively.

As shown in Fig 5b, in the second embodiment, using P impurities, a 10 channel monolithic multiple wavelength laser structure was achieved. The wavelengths were 1.557  $\mu$ m, 1.555  $\mu$ m, 1.550  $\mu$ m, 1.548  $\mu$ m, 1.543  $\mu$ m, 1.530  $\mu$ m, 1.514  $\mu$ m, 1.487  $\mu$ m, 1.479  $\mu$ m and 1.474  $\mu$ m respectively.

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The reason for not achieving 10-channels in the first embodiment is because the gray levels were found to saturate outside the gray 3 to gray 8 region. This explains the reason why certain channels give similar lasing wavelength after quantum well intermixing.

After measuring the light-current characteristics of the As\*\* and P\*\* multiple wavelength lasers, the threshold currents and the slope efficiencies were then analysed (Fig. 4c and 5c).

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For the first embodiment,  $As^{**}$ -IID lasers fabricated from regions with  $SiO_2$  completely removed suffered from a 25 % increase in threshold current, from 1.6 kA/cm² to 2 kA/cm² (Figure 4c).

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For the second embodiment, only a 17 % increase of threshold current density, from 1.2 kA/cm² to 1.4 kA/cm² was observed between the non-implanted (gray 1: full oxide, non-intermixed but annealed) and direct implanted (gray 10: no oxide, fully intermixed and annealed) regions -- see Figure 5c.

However, for both embodiments, the slope efficiency shows very little change. This indicates that the quality of the materials remains high after intermixing using the technique of the invention.

The method of the invention may be used to produce other components as well as multi-frequency devices because the material produced is of high electrical and optical quality. Thus the technique is suited to the production of photonic integrated circuits.

Although the present invention has been described using a specific compound semiconductor, it is applicable to other compound semiconductors. The embodiment has been described using oxide as the mask but it will be

understood by those skilled in the art that any suitable dielectric can be used or indeed that a polymer or metal mask can be used instead. In the described embodiment a mask was used as well as a photoresist but it will also be clear to those skilled in the art that a graded photoresist could be used without an additional mask. The embodiment has been described in the context of a rapid thermal processor but rapid thermal annealers, furnaces and other temperature devices may be used instead.

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It is also understood by those skilled in the art that routine practices including the following can be applied to enhance the performance and functionality of the said multi-wavelength laser diodes:-

- optical gratings can be incorporated in the laser structure to provide distributed feedback to the laser arrays, and also to provide fine-tuning of the lasing wavelengths to specified values. Such distributed feedback lasers or distributed Bragg reflecting lasers allows for an improved narrow spectral line-width and good side-mode suppression.
- The optical outputs from the multi-wavelength laser arrays can be combined together in an integrated passive coupler or monolithic multimode-interference combiner for transmission into an optical modulator, optical amplifier, or launching into an optical fiber.
- 30 3) Optical coatings of specified reflectivity could be applied to the facets of the device to enhance its output efficiency.

Although the described embodiments relate to laser diode arrays emitting around 1.5  $\mu m$ , the process technology can also be used to form devices operating at other wavelengths. It is understood therefore that the structures of the invention are not restricted to any specific wavelength.

#### CLAIMS:

1. A laser structure comprising a plurality of quantum well regions on a semiconductor substrate, wherein each said region contains a respective different concentration of an impurity, to thereby provide a corresponding plurality of different radiation wavelengths in use.

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- 2. The laser structure of claim 1 wherein said substrate is a III-V semiconductor material.
- 3. The laser structure of claim 2 wherein said material comprises InGaAs.
  - 4. The laser structure of any preceding claim wherein said impurity comprises As.
- 20 5 The laser structure of any of claims 1-3 wherein said impurity comprises P.
- 6. The laser structure of any preceding claim wherein each said region comprises a quantum well intermixed region.
  - 7. An integrated circuit comprising the structure of any preceding claim, and electrical connections for applying respective pumping signals to said regions.

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- 8. A method of manufacturing a laser structure capable of emitting different radiation wavelengths comprising providing a semiconductor substrate, forming a quantum well region therein, characterised by: differentially masking portions of said region, implanting impurities into said differentially masked portions, and annealing said structure whereby each said portion in use emits a respective one of said different radiation wavelengths.
- 10 9. The method of claim 8 wherein said step of differentially masking portions of said region comprises forming a masking layer on said portion of said region and differentially etching said masking layer.
- 15 10. The method of claim 9 wherein before said etching step, the method comprises forming a photoresist on said masking layer, applying masks having different optical densities to said photoresist and developing said photoresist.

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- 11. The method of claim 10 wherein said step of forming a photoresist comprises spin-coating said photoresist on said masking layer.
- 25 12. The method of any of claims 9-11 wherein said masking layer comprises a dielectric.
  - 13. The method of claim 12 wherein said dielectric is silicon dioxide.

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14. The method of any of claims 9-11 wherein said masking layer comprises a polymer.

- 15. The method of any of claims 9-11 wherein said masking layer comprises a metal.
- 5 16. The method of claim 9 wherein said etching step comprises dry etching.
- 17. The method of claim 16 wherein said dry etching has a one-to-one selectivity between photoresist and said masking layer.
  - 18. The method of claim 17 wherein process parameters of a dry etching system are selected to provide said one-to-one selectivity.
  - 19. The method of claim 8 or claim 9 wherein said step of differentially masking portions of said region comprises using a graded photoresist.

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- 20 20. The method of any of claims 8-19 wherein said step of implanting impurities comprises ion implantation.
- 21. The method of any of claims 8-19 wherein said step of implanting impurities comprises a focused ion beam 25 technique.
  - 22. The method of any of claims 8-19 wherein said step of implanting impurities comprises diffusion.
- 30 23. The method of any of claims 8-22 wherein said annealing step is performed in a rapid thermal processor.

- 24. The method of any of claims 8-23 wherein said annealing step uses a rapid thermal annealer.
- 25. The method of any of claims 8-22 wherein said annealing step is performed in a furnace.
  - 26. The method of any of claims 8-25 wherein said impurities comprise phosphorus.
- 10 27. The method of any of claims 8-25 wherein said impurities comprise arsenic.

#### ABSTRACT

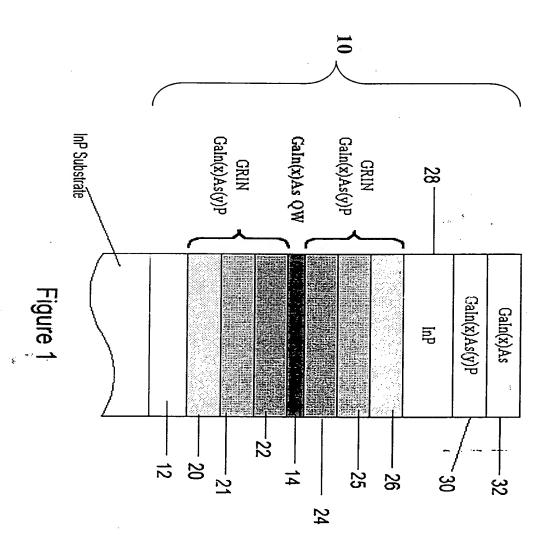
#### MULTIPLE WAVELENGTH LASERS

A multiple wavelength laser has plural quantum well regions on a substrate, each region containing a respective different concentration of an impurity to provide plural radiation wavelengths.

An impurity induced disordering technique using a gray mask is also described.

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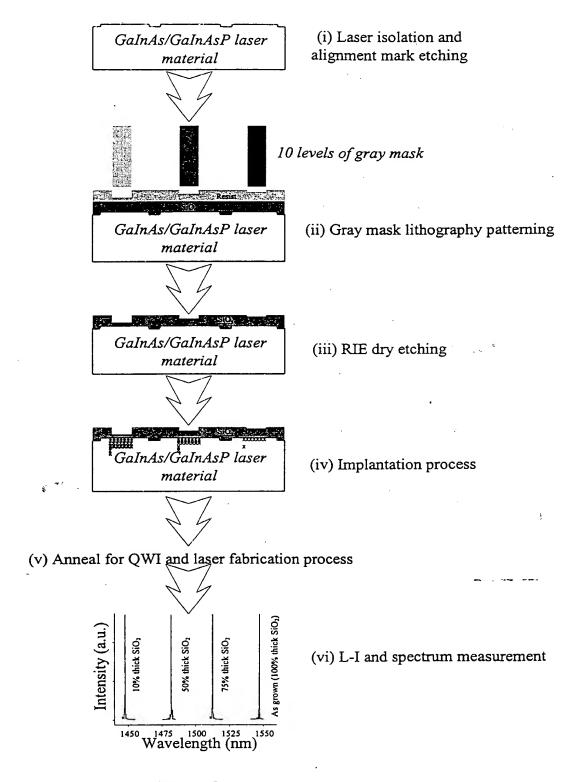


Figure 2

- ---Photoresist thickness before etching
- --- SiO₂ thickness after etching

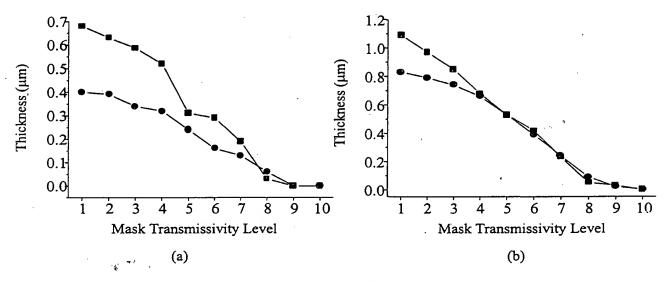


Figure 3

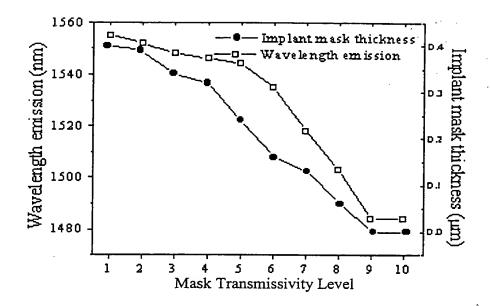


Figure 4(a)

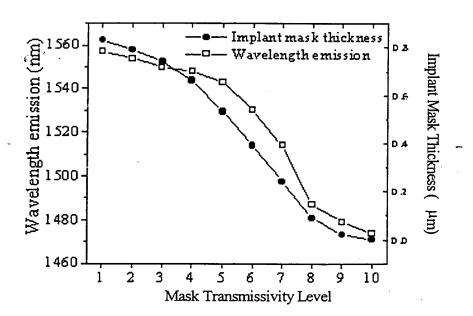


Figure 5(a)

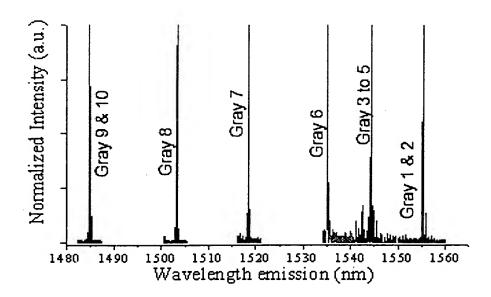


Figure 4(b)

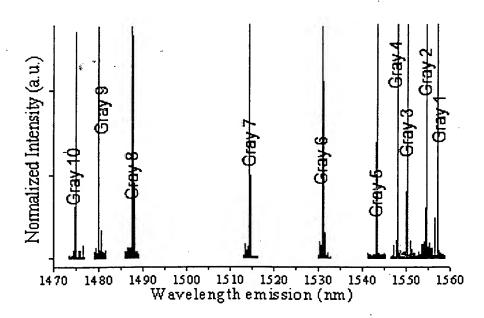


Figure 5(b)

